1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples are filtered through 0.45 µm membrane filters and acid-preserved prior to analysis. No digestion is needed prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is performed for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are needed. Acid digestion is also needed prior to analysis to dissolve elements in drinking water samples with turbidity <1 NTU. This SOP follows the guidelines of the EPA Methods 200.8 and 6020.

1.2 The metals that can be determined by using this SOP are listed bellow. Elements specific to each method (*i.e.* 6020, 6020A, 200.8) and reporting limits are listed in Table 1.

Metal (Symbol)	CAS#:
Aluminum (Al)	7429-90-5
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-2
Barium (Ba)	7440-39-3
Beryllium (Be)	7440-41-7
Boron (B)	7440-42-8
Cadmium (Cd)	7440-43-9
Calcium (Ca)	7440-70-2
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Iron (Fe)	7439-89-6
Lead (Pb)	7439-92-1
Lithium (Li)	7439-93-2
Magnesium (Mg)	7439-95-4
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-95-4
Nickel (Ni)	7440-02-0
Potassium (K)	7440-09-7
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Sodium (Na)	7440-23-5
Strontium (Sr)	7440-24-6
Tin (Sn)	7440-31-5
Titanium (Ti)	7440-32-6
Thallium (Tl)	7440-28-0
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

1.3 If this SOP is used to determine any analyte not listed in the table above, it is the responsibility of the analyst to demonstrate the accuracy and precision of the Method in the samples to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

1.4 Use of this method is restricted to analysts who are knowledgeable in the recognition and in the correction of spectral, chemical and physical interferences in ICP-MS.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples which require total ("acid-leachable") values are digested using appropriate sample digestion procedures (see SOP 3015dig and 3051dig).

2.2 This SOP describes the measurement of ions produced by radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios, and quantified with a channel electron multiplier. Potential interference from isobaric elements and polyatomic ions are corrected for by the use of elemental interference equations based on natural isotope abundance. Interference corrections include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. Instrument drift and matrix induced signal suppressions and enhancements are compensated for by the use of internal standardization.

3.0 INTERFERENCES

There are three fundamentally different sources of interference in ICP-MS: spectroscopic interferences, physical, and memory interferences.

3.1 **Spectroscopic Interferences** are interferences caused by the presence of compounds or elements entering the mass spectrometer which have the same nominal mass to charge (m/z ratio as the analyte elements. They can be isobaric elemental and isobaric molecular interferences (polyatomic, refractory oxide, and doubly charged ions).

3.1.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z) as the analyte element. These can be managed by the selection of an alternate isotope for analysis or by the use of elemental interference equations. These equations use the naturally occurring isotope ratios of most elements to estimate and allow for the subtraction of isobaric interferences. An example of an elemental isobaric interference is ^{40}Ar on ^{40}Ca. In this case, the use of ^{43}Ca or ^{44}Ca is recommended. The appropriate elemental interference equations are incorporated in the methods (or parameter) used for calibration and data acquisition.

3.1.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundance, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1%) counting statistics.

3.1.2.1 Example for As is as follows: Because the ^{35}Cl natural abundance of 75.77 % is 3.13 times the ^{37}Cl abundance of 24.23 %, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 % of the $^{40}Ar^{35}Cl^+$ signal): Corrected arsenic signal (using natural isotopes abundance for coefficient approximations) = (m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal), (where the final term adjusts for any selenium contribution at 77 m/z).

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes[6]).

3.1.2.2 Example for Cd is as follows: corrected cadmium signal (using natural isotopes abundance for coefficient approximations) = (m/z 114 signal) - (0.027)(m/z 118

signal) - (1.63)(m/z 108 signal), (where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114).

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: Since there is a certain degree of uncertainty as to which equation is better to use, and in what cases, it is up to the analyst to determine how the interference will be corrected, upon the evaluation of data. It is suggested that the elemental isobaric interference equations be included in all methods (parameters) from the beginning, but potential polyatomic species (masses) that could interfere be only monitored (except for $^{40}Ar^{35}Cl^+$ on As). When species monitored indicate that an isobaric molecular interference is present, the equations can be adjusted to correct for such interference, and data be reprocessed to produce an interference free summary report. Generally, an interference is easy to spot when multiple isotopes of an element show different results. Since the interference is additive, the use of the isotope with the lowest result is suggested for data reporting, providing that all other QC criteria are met.

3.1.3 Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. Wing overlap interference may occur when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized, and the spectrometer resolution adjusted to minimize them. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require matrix separation, or analysis using another verified and documented isotope.

3.2 **Physical Interferences** are associated with the physical processes, which govern the transport of sample into the plasma, sample conversion process within the plasma and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the samples and calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g. viscosity effects), at the point of aerosol formation and transport to the plasma (e.g. surface tension effects), during the atomization and ionization process within the plasma itself, or during the transfer of ions through the interface and mass spectrometer (space charge effects). To minimize some of these effects, acid composition and concentration should be matched for all standards, blanks, and samples. Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards should ideally display similar analytical behavior to the elements being determined. Generally, an internal standard should be no more than 50 amu removed from the analyte. Recommended internal standards include ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{169}Ho, ^{185}Re, and ^{209}Bi.

3.3 **Memory Interferences** result when elements in a previous sample contribute to signals measured in a subsequent sample. Memory effects can result from the deposition of sample on various components of the sample introduction system, including sample and peristaltic pump tubing, spray chamber, torch, and interface cones. The site(s) where deposition may occur is dependent on the sample and may need to be minimized through the use of a rinse blank between samples. Routine maintenance (cleaning and/or replacement) of sample introduction components is necessary for long-term minimization of memory effects. The possibility of memory interferences within an analytical run should be recognized and suitable rinse times should be used to reduce them. Memory effects are evaluated by using a minimum of three replicate integrations for data acquisition. High relative standard deviation (%RSD) of the three replicates caused by a consecutive drop in signal intensity is indicative of carryover from the previous sample. If a memory interference is suspected, the sample should be reanalyzed after analysis of a blank indicates that the carryover has been eliminated

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled plasma-mass spectrometers:

4.1.1 **Perkin Elmer (PE) Elan 9000 ICP-MS:** :
- Windows XP Operating System
- Elan 3.0 Software
- Cetac Asx510 Autosampler

4.1.2 **Hewlett-Packard (HP) 4500 ICP-MS**:
- Windows 95 operating system.
- Chem Station Software.
- Cetac ASX150 Autosampler.

4.2 Argon gas supply: liquid argon cylinders.
4.3 Analytical balance, 510gcapacity, minimum accuracy ± 0.001g.
4.4 Digital bottle top dispenser capable of dispensing volumes of 0-5 ml in 0.02 ml increments.
4.5 Eppendorf automatic pipette with disposable combitips ranging from 2.50 ml to 50 ml capable of pipetting volumes ranging from 50 µl to 5,000 µl.
4.6 Disposable Pasteur pipettes.
4.7 Polypropylene vessels, 50 mL.
4.8 Plastic cups to support minimum of 200 ml.
4.9 Plastic bottles.

5.0 REAGENTS

5.1 Nitric Acid (HNO_3), concentrated, Trace Metal Grade. Acids used in the preparation of standards and for sample processing must be of high purity. Trace metal grade (also known as re-distilled) acids are recommended because of the high sensitivity of ICP-MS. Nitric acid at 2% (v/v) or less in the solution to be analyzed is required for ICP-MS, in order to minimize damage to the interface.

5.2 Hydrochloric Acid (HCl), concentrated, Trace metal Grade. Several polyatomic ion interferences result when HCl is used. However, its use is recommended to maintain stability in solutions containing high concentrations of antimony and silver. When used, corrections for the chloride polyatomic ion interference must be applied to all data.

5.3 Reagent water (Deionized water): All references to reagent water in the method refer to ASTM Type I water (ASTM D1193), unless otherwise specified.

5.4 Internal Standard stock solutions:

5.4.1 Lithium 6, 1000 µg/ml stock solution.
5.4.2 Scandium, 1000 µg/ml stock solution.
5.4.3 Yttrium, 1000 µg/ml stock solution.
5.4.4 Rhodium, 1000 µg/ml stock solution.
5.4.5 Rhenium, 1000 µg/ml stock solution.

5.4.6 Internal Standard working solution (**IS-WS**): From the above stock solution, 5 g of each is transferred to a 1000 ml plastic bottle, along with 20 ml of concentrated HNO_3 and brought to a final volume of 1000 ml (by weight). The concentration in the flask will be approximately 5.0 µg/ml. This represents the internal standards working solution from which 1 ml (for a 50 ml final volume) will be added to all calibration standards and blanks. This will provide a 0.10 ppm of internal standard concentration in all calibration standards, similar to analytical samples.

Note: The stock solutions are NIST traceable, and provided with a certificate of analyses and MSDS sheets by the vendor.

5.5 Multielement standard stock solution from three different vendors:

5.5.1 Inorganic Venture (IV), 100mg/L each of Ag, Al, B, Ba, As, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Ti, Tl, V, Zn.. This solution is used for the preparation of standards.

5.5.2 High Purity Standards (**HM**), 100 µg/ml each of Ca, Mg, K, Na. This solution is used for the preparation of the calibration standards and for the minerals standard.

5.5.3 SPEX Industries (**S**), 100 µg/ml each of Al, Sb, As, Ba, Be, B, Cd, Ca Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sr, Sn, Ti, Tl, V, Zn. This solution is used for the preparation of the initial calibration verification (ICV) standards.

5.5.2 Environimental Resourse Associates (ERA), concentration varies by lot number and element but contains all of the following Al, Sb,As,Ba, Be, B Cd, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Se, Ag, Zn, V, Th, S. This solution is used as a third source of verification

Note: The stock solutions are NIST traceable, and provided with a certificate of analyses and MSDS sheets by the vendor. See Appendix 1 for standard preparation.

5.6 Multielement calibration standard solutions are prepared by diluting the stock standard solutions to levels in the linear range for the instrument in a solvent consisting of 2% (v/v) HNO_3 in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. The calibration standards are kept in plastic bottles, and prepared every two weeks or as needed. They must be verified using a quality control standard (ICV). Table 2 and Table 3 can be used as guidance, when preparing standards.

5.7 Blanks: Three types of blanks are required for the analysis. The calibration blank (std-0.00) is used in establishing the calibration curve. The preparation blank (LRB) is used to monitor possible contamination resulting from the sample preparation procedure. The rinse blank (also called optional rinse or autosampler wash) is used to flush the system between all samples and standards.

5.7.1 The calibration blank (std-0.00) and the continuing calibration blank (CCB) consists of the same concentration(s) of the same acid(s) used to prepare the calibration standards, along with the appropriate concentration of internal standard.

5.7.2 The preparation (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions.

5.7.3 The rinse blank consists of 2% HNO_3 (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

5.8 The interference check solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interference such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

5.8.1 Interference check stock solution A, containing 1000 µg/ml each of Al, Ca, Fe, Mg, Na, P, K, S, 2000 µg/ml of C, 10000 µg/ml of Cl, and 20.0 µg/ml each of Mo and Ti. The ICS-A solution is prepared by weighing 10.0 g of the stock solution in a plastic cup, addition of 2 ml IS-WS, 2ml HNO_3, and dilution to 100 g on the scale with reagent water.

5.8.2 Interference check stock solution AB, containing 2.0 µg/ml each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn. The ICS-AB solution is prepared by weighing 1.00 g of the stock solution in a plastic cup, addition of 2 ml IS-WS, 2ml HNO_3, and dilution to 100 g on the scale with reagent water.

5.8.3 The final concentration of the elements in ICS-A and ICS-AB is listed in Table 5. These solutions are prepared fresh every two weeks or as needed.

5.9 The quality control standard is the initial calibration verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near the midpoint of the linear range at a concentration other than that used for instrument calibration. An independent standard is defined as a standard from a source different from those used in the standards for instrument calibration.

5.10 Mass spectrometer tuning solution. A solution containing elements representing all of the mass regions of interest must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications (see Section 7.5). This solution is also used to verify that the instrument has reached thermal stability (See Section 7.4).

5.10.1 Tuning solution for HP 4500 ICP-MS: 10 µg/ml each of Li, Y, Ce, and Tl. Take 1.00 g of this solution in a plastic bottle, add 20 ml HNO_3, and dilute to 1000 g on the scale with reagent water (or add 979 g reagent water to the bottle with standard and acid). This will result in a 10.0 ppb solution of the above elements, used to tune the instrument according to the manufacturer instructions.

5.10.2 Tuning solution for PE Elan 9000 Tune Solution 6020-Li Co In Tl (10 ppb) Tune Solution 200.8- Be Mg Co In Pb (10ppb)This is used to tune the instrument according to the manufacturer instructions.

5.11 Drinking water working standards are prepared every two weeks

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples are collected in appropriate containers. For water samples, the samples are collected in HNO_3 pre-preserved plastic container (approximately 125 ml volume), and are acidified to pH of <2.

6.2 Soil samples are collected without preservation, usually in glass containers with Teflon lined caps. Non-aqueous samples should be refrigerated upon receipt and analyzed as soon as possible.

6.3 Holding times for metals are 6 months from the date of sampling.

7.0 PROCEDURE

7.1 Solubilization and digestion procedures are presented in the Sample Preparation SOP's (e.g., 3015dig, 3051dig).

7.2 Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions.

7.3 Set up the instrument with the proper operating parameters according to the instrument manufacturer's instructions (Table 4).

7.4 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. This must be verified by analyzing a tuning solution (Section 5.10.1 and 5.10.2).

7.5 Tune the instrument according to the instrument manufacturer's instructions. For drinking water samples the tuning requirements are listed in the 200.8 method. The tuning should include beryllium, magnesium, cobalt, indium, and lead. Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria that must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be within 0.5-0.9 amu full width at 10 percent peak height.

NOTE: Precautions must be taken to protect the channel electron multiplier from high ion currents. The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this time period, response factors are constantly changing, which invalidates the calibration curve, causes instability, and invalidates sample analyses.

7.6 Calibrate the instrument for the analytes of interest (recommended isotopes for the elements in Section 1.2 are provided in Table 6a and Table 6b), using the calibration blank and at least a single initial calibration standard according to the instrument manufacturer's procedure. Table 2 and Table 3 (Section 5.6) provides information as to what calibration standards to use. Flush the system with the rinse blank (Section 5.7.3) between each standard solution. Use the average of at least three integrations for both calibration and sample analyses.

7.7 All masses that could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 6a, and Table 6b).

7.8 Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the calibration verification solution (ICV, Section 5.9). When measurements exceed ± 10% of the accepted value, the analyses must be terminated, the problem corrected, the instrument re-calibrated, and the new calibration verified. During the course of an analytical run, the instrument may be "re-sloped" or re-calibrated to correct for instrument drift. A re-calibration must then be followed immediately by a new analysis of a ICV and ICB before any further samples may be analyzed. Corrective actions for specific situations are summarized in Table 7.

7.9 An optional Blank Spike (BS) of low concentration can be used to verify the linearity of the calibration curve near the lower end. When such standard is used, recalibrate the instrument if the recovery of the BS is outside 70-130% of true concentration.

7.10 Flush the system with the rinse blank solution (Section 5.7.3) until the signal levels return to the method's levels of quantitation (usually about 30 seconds) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (usually about 30 seconds) prior to collecting data. Analyze the calibration verification solution (CCV), and the continuing calibration blank (CCB) at a frequency of at least once every 10 analytical samples.

7.11 Dilute and reanalyze samples that are more concentrated than the linear range (LDR Section 8.2.2.) for an analyte or measure an alternate less-abundant isotope. The linearity at the alternate mass must be confirmed by appropriate calibration (see Sec. 7.6 and 7.8).

7.12 Calculations: The quantitative values shall be reported in appropriate units, such as milligrams per liter (mg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples.

7.12.1 The appropriate dilution factor (DF) resulted from sample preparation (see 3015dig and 3051B SOP's) is entered in the data system for each sample at the time of programming the

sequence to be analyzed. If additional dilutions are performed, the appropriate corrections must be applied to the dilution factor.

7.12.2 Generally, for solid samples, DF includes the correction necessary for the determination of a dry weight result. If this is not the case or if a dry weight result is requested at a later time, calculate results for solids on a dry-weight basis as follows:

(1) A separate determination of percent solids must be performed.
(2) A new DF can be calculated, based on the original wet weight of the sample (from the preparation log) and the percent total solids. The sequence is updated with the new DF, data reprocessed, and a new quantitation report is generated by the data system.
(3) Manual calculation of the dry weight concentration (DWC) by the formula:

$$DWC\ (mg\ /\ kg) = \frac{CxV}{WxS}$$

Where, C = Digest Concentration (mg/L).
V = Final volume in liters after sample preparation.
W = Weight in kg of wet sample.
S = (% Total Solids)/100.

7.12.3 Calculations performed by the data system include appropriate interference corrections, internal-standard normalization, and the summation of signals at 206, 207 and 208 m/z for lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and be available for easy reference or inspection.

8.2 Initial Demonstration of Performance.

8.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges) and laboratory performance (determination of method detection limits) prior to analyses conducted by this method.

8.2.2 Linear calibration ranges: Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range, which may be used for the analysis of samples, should be judged by the analyst from the resulting data. The upper LDR is defined as the maximum concentration for which the measured concentration is within ±10% of the true value. Determined sample analyte concentrations that are greater than the upper LDR limit must be diluted and reanalyzed. The LDR should be verified whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.2.3 Method detection limits (MDL) should be established for all analytes, using reagent water (blank) fortified at a concentration of two to five times the estimated detection limit. To determine MDL values, take seven 7 replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

MDL = (t) x (S)

Where: t = student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (t = 3.14 for seven replicates);
S = standard deviation for the replicate analyses.

MDL's should be determined annually, when a new operator begins work or whenever, in the judgement of the analyst, a change in the instrument hardware or operating conditions would dictate they be redetermined.

8.3 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantification or the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferant itself, but that a molecular species may be monitored to indicate the presence of the interferent. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon, and sulfur molecular ions could impact the analytes of interest. Unsuspected interferences may be detected by adding pure major matrix components to a sample to observe any impact on the analyte signals. If an interference source is present, and can not be corrected, the sample elements impacted must be flagged. When correction equations are used, all QC criteria must also be met.

8.4 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall outside limits as compared with the first calibration standard (Calibration Blank or std-0.00), the following procedure is followed. The sample must be diluted at least fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal-standard intensities fall within the prescribed window. The intensity levels of the internal standards for the ICV/ICB, CCV/CCB, LCS/LRB must also be within the specified acceptance limits (refer to Section 8.9.1.3 and 8.9.2.4 for limits). If they are not, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.5 Check the instrument calibration by analyzing appropriate quality control solutions as follows:

8.5.1 Check instrument calibration by analyzing the initial calibration verification solution (ICV) and the initial calibration blank (ICB).

8.5.2 Verify calibration at a frequency of every 10 analytical samples with the CCV standard and the continuing calibration blank (CCB). These solutions must also be analyzed for each analyte at the beginning of the analysis and after the last sample.

8.5.3 The results of the ICV and CCV must agree within ± 10% of the expected value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.5.4 The results of the ICB and CCB's must be less than the current RDL for each element or less than the reporting limits for sample, whichever is greater. If this is not the case, the reason for the out-of-control condition must be found and corrected, and affected samples must be reanalyzed.

8.6 A Laboratory Control Sample (LCS) should be analyzed for each analyte using the same sample preparations, analytical methods, and QA/QC procedures employed for the test samples. One LCS should be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less. The recovery limits for the LCS are 85-115% of the true value (stated in the preparation log).

8.7 Analyze one matrix spike (MS) sample for every 10 analytical water samples or every 20 analytical soil samples. For majority of the elements, the aqueous samples are spiked at levels similar to the LCS (0.05 ppm in the analysis solution). For solid samples, the concentration added is approximately 20 mg/Kg equivalent (0.10 ppm in the analysis solution). The acceptable limits for performance are summarized in Section 8.10.

8.7.1 Calculate the percent recovery of each analyte, corrected for background concentrations measured in the unfortified (original) sample. Percent recovery may be calculated in units appropriate to the matrix, using the following equation:

$$R = \frac{C_s - C}{S} x100$$

where: R = percent recovery.
C_s = spiked sample concentration.
C = sample background concentration.
S = concentration equivalent of analyte added to fortify the sample.

8.8 Analyze one matrix duplicate (Dp) sample for every 10 water samples or every 20 soil samples. In some cases, a matrix spike duplicate (MSD) can be used instead of the matrix duplicate, especially if the analytes in the sample are of low concentration. A control limit of 20% RPD should not be exceeded for analyte values greater than 100 times the MDL. If this limit is exceeded and laboratory performance for that analyte is shown to be in control (ICV/ICB, CCV/CCB, and LCS/LRB within the limits), the problem encountered is judged to be matrix related. The data user should be informed that the result for that analyte is suspect due to the heterogeneous nature of the sample. If the performance of the laboratory is not in control (ICV/ICB, CCV/CCB, and LCS/LRB outside the limits), the reason for the out-of-control situation must be found and corrected, and any samples analyzed during the out-of-control condition for that analyte must be reanalyzed.

8.8.1 The relative percent difference (RPD) between duplicate determinations must be calculated as follows:

$$RPD = \frac{\left| D_1 - D_2 \right|}{\frac{D_1 + D_2}{2}} x\,100$$

where: RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate).

8.9 The Quality Control requirements and limits vary slightly, based upon the method referenced in the analytical report (i.e. 6020 vs. 200.8). For both methods, the calibration is verified by the analysis of ICV/ICB and CCV/CCB. Recalibration is required when either one falls outside the limits. The performance of the method is evaluated by the analysis of the LCS/LRB pair for every batch of 20 samples, and MS/MSD/Dp for every 10 samples.

8.9.1 Method 200.8 specific requirements:

8.9.1.1 When the recovery for ICV/CCV falls outside ±10% terminate the analysis and recalibrate the instrument. If the last CCV was within 15% of the true concentration, the results for the samples are still acceptable. If this is not the case, the only acceptable results are the once corresponding to samples analyzed before the last CCV that was within 15% of the true concentration. All other samples are to be analyzed again, after recalibration of the instrument.

8.9.1.2 The recovery limits for MS samples are 70-130%. If the recovery of any analyte falls outside the designated range and the laboratory performance is shown to be in control (ICV/ICB, CCV/CCB, and LCS/LRB within the limits), the recovery problem encountered with the spiked sample is judged to be matrix related, not system related. The data user should be informed that the result for the analyte in the unspiked sample is suspect due to an uncorrected matrix effect. Recovery is not required if the concentration of the analyte added is less than 30% of the concentration of the analyte in the original sample.

8.9.1.3 The absolute response of any one internal standard must not deviate by more than 60 to 125% of the original response in the first calibration standard (Calibration Blank or std-0.00). If deviations greater than these are observed flush the instrument with rinse blank, than analyze a CCB. If the responses of the internal standards are now within the limit proceed with sample dilution as described in Section 8.4. If the responses of the internal standards are not within the limit, terminate the analysis, recalibrate the instrument, and reanalyze the samples from the last CCB with acceptable internal standard recoveries.

8.9.2 Method 6020 specific requirements:

8.9.2.1 When the recovery for ICV/CCV falls outside ±10% terminate the analysis and recalibrate the instrument. The samples from the last CCV that was within limits are to be re-analyzed, after recalibration of the instrument.

8.9.2.2 The MS is represented by a spiked sample, before digestion, and a Post-Digestion Spike, if the recovery of the regular spike fails to meet QC criteria. An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 percent of the known value. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the original sample must be diluted to compensate for the matrix effect, and reanalyzed, after a new post-digestion spike is added. The same recovery limits apply to the spiked dilution.

8.9.2.3 The MSD is represented by a spiked duplicate sample, before digestion, and a Post-Digestion Spike, if the recovery of the regular duplicate spike fails to meet QC criteria. The evaluation of the MSD is similar to the evaluation of the duplicate analysis described in Section 8.8.

8.9.2.4 When the intensity of any internal standard in the sample to falls outside 30-120% of the intensity of that internal standard in the initial calibration standard (Calibration Blank or std-0.00), follow the procedure described in Section 8.4. The intensity levels of the internal standards for the ICV/ICB and CCV/CCB must agree within ± 20 percent of the intensity level of the initial calibration standard (Calibration Blank or std-0.00). If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.9.2.5 Dilution Test: If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 x MDL), an analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected, and the results flagged. One dilution test must be included with every batch of twenty samples.

8.9.2.6 Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the interference check solutions ICS-A and ICS-AB. The recovery of the elements of interest in ISC-AB (listed in Table 5 at a concentration of 0.02 ppm) should be between 70-135%.

8.10 Summary of the QC requirements and performance acceptance limits are shown in the following table:

QC Type	Method 6020 (%)	Method 200.8 (%)
ICV	90-110	90-110
BS	70-130	70-130
CCV	90-110	90-110*

Dp	0-20	0-20
MS	75-125	70-130
MSD	0-20	0-20
ICS-AB	70-135	n.a.
Internal Standard	80-120 for CCV/CCB 30-120 for samples	80-120 for CCV/CCB 60-125 for samples

*NOTE: *Sample results still acceptable if CCV between 85-115%.*

9.0 METHOD PERFORMANCE

9.1 The precision and accuracy of the method will depend upon the overall performance of the sample preparation and analysis.

9.2 Performance Evaluation samples are analyzed periodically in order to prove the performance of the method.

9.3 In an EPA multi-laboratory study, laboratories applied the ICP-MS technique to both aqueous and solid samples. The results are listed at the end of methods 200.8 and 6020.

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11.0 APPROVAL & ISSUE:

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Paul Roettger, Senior Analyst Date

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Andrew Ball, QA Officer Date

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Maya V. Murshak, Technical Director Date

12.0 LIST OF TABLES

Table 1. List of elements analyzed.

Element	Symbol	CAS#	Reporting Limits		Method 6020	Method 200.8
			mg/L	mg/Kg		
Aluminum	(Al)	7429-90-5	0.05	0.5	Aluminum	Aluminum
Antimony	(Sb)	7440-36-0	0.002	0.3	Antimony	Antimony
Arsenic	(As)	7440-38-2	0.001	0.1	Arsenic	Arsenic
Barium	(Ba)	7440-39-3	0.01	1.0	Barium	Barium
Beryllium	(Be)	7440-39-3	0.001	0.50	Beryllium	Beryllium
Boron	(B)	7440-42-8	0.01	1.0	-	-
Cadmium	(Cd)	7440-43-9	0.0005	0.2	Cadmium	Cadmium
Calcium	(Ca)	7440-70-2	0.05	10.0	Calcium	-
Chromium	(Cr)	7440-47-3	1.0	2.0	Chromium	Chromium
Cobalt	(Co)	7440-48-4	0.005	2.0	Cobalt	Cobalt
Copper	(Cu)	7440-50-8	0.01	0.5	Copper	Copper
Iron	(Fe)	7439-89-6	0.004	1.0	Iron	-
Lead	(Pb)	7439-92-1	0.1	1.0	Lead	Lead
Lithium	(Li)	7439-93-2	0.003	1.0	-	-
Magnesium	(Mg)	7439-95-4	0.01	1.0	Magnesium	-
Manganese	(Mn)	7439-96-5	1.0	4.0	Manganese	Manganese
Molybdenum	(Mo)	7439-98-7	0.02	1.0	-	Molybdenum
Nickel	(Ni)	7440-02-0	0.005	0.50	Nickel	Nickel
Potassium	(K)	7440-09-7	0.005	1.0	Potassium	-
Selenium	(Se)	7782-49-2	1.0	5.0	Selenium	Selenium
Silver	(Ag)	7440-22-4	0.005	0.2	Silver	Silver
Sodium	(Na)	7440-23-5	0.0002	0.1	Sodium	-
Strontium	(Sr)	7440-24-6	0.05	10.0	-	-
Tin	(Sn)	7440-31-5	0.005	0.50	-	-
Titanium	(Ti)	7440-32-6	0.02	1.0	-	-
Thallium	(Tl)	7440-28-0	0.005	1.0	-	-
Vanadium	(V)	7440-62-2	0.001	0.50	Thallium	Thallium
Zinc	(Zn)	7440-66-6	0.004	1.0	Vanadium	Vanadium
			0.01	1.0	Zinc	Zinc

Table 2. Standard preparation for all elements except Ca, Mg, K, Na.

STANDARD PREPARATION

Inorganic Stock Solution IV-7 + IV-19 (100 ppm)

I. Working Stock Solution 1 (WS1)

10 mls IV-7 + 10 mls IV-19 + 2 mls HNO_3; Bring to a final volume of 100 mls = 10 ppm

II. Working Stock Solution 2 (WS2)

1ml of WS1 to a final volume of 100 mls = 0.100 ppm

III. Standards

Standards	Volume Working Stock Solution	Internal Standard 5.0 ppm	HNO_3	1.1. Final Volume
0.20 ppm	2.00 mls WS1	2 mls	2 mls	100 mls
0.10 ppm	1.00 ml WS1	2 mls	2 mls	100 mls
0.05 ppm	0.50 ml WS1	2 mls	2 mls	100 mls
0.02 ppm	20.0 mls WS2	2 mls	2 mls	100 mls
0.01 ppm	10.0 mls WS2	2 mls	2 mls	100 mls
0.005 ppm	5.00 mls WS2	2 mls	2 mls	100 mls
0.002 ppm	2.00 mls WS2	2 mls	2 mls	100 mls
0.0005 ppm	0.50 mls WS2	2 mls	2 mls	100 mls

Table 3. Standard preparation for Ca, Mg, K, Na.

Standards **Ppm**	**Volume of Working Stock**	**Source and Lot # of Working Stock (100ppm)**	**Internal Standard 5.0 ppm**	**HNO_3**	Final Volume
0.50	**0.5**	**High Purity - 620719**	**2 mls**	**2 mls**	**100 mls**
1.0	**1.0**	**High Purity - 620719**	**2 mls**	**2 mls**	**100 mls**
2.0	**2.0**	**High Purity - 620719**	**2 mls**	**2 mls**	**100 mls**
5.0	**5.0**	**High Purity - 620719**	**2 mls**	**2 mls**	**100 mls**
10.0	**10.0**	**High Purity - 620719**	**2 mls**	**2 mls**	**100 mls**
ICV- 5.0	**5.0**	**Spex – 5-59JB**	**2 mls**	**2 mls**	**100 mls**

Table 4. ICS Components and Concentration.

Solution Component	**ICS-A (ppm)**	**ICS-AB (ppm)**
Al	100.0	100.0
Ca	100.0	100.0
Fe	100.0	100.0
Mg	100.0	100.0
Na	100.0	100.0
P	100.0	100.0
K	100.0	100.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.0
Ti	2.0	2.0
As	0.0	0.020
Cd	0.0	0.020
Cr	0.0	0.020
Co	0.0	0.020
Cu	0.0	0.020
Mn	0.0	0.020
Ni	0.0	0.020
Ag	0.0	0.020
Zn	0.0	0.020

Table 5a. Recommended analytical isotopes (underlined) and additional masses to be monitored.

Mass	**Element**	**I.S. Used**	**Elemental Correction**	**Potential interferences**
19	K	Sc, Rh		
23	Na	Sc, Rh		

24	Mg	Sc, Rh		
43	Ca	Sc, Rh		
44	Ca	Sc, Rh	$(-0.0271)(^{88}C)$	Sr^{++}

Notes:
- † Recommended for PE instrument.
- ‡ Recommended for HP instrument.
- C = Counts at specified mass.
- When the concentration of Na in the samples is high, the ionization of Sc is suppressed leading to positive bias of the results, therefore Rh should be used as the internal standard, even if more than 50 amu removed from the element of interest.

Table 5b. Recommended analytical isotopes (underlined) and additional masses to be monitored.

Mass	Element	I.S. Used	Elemental Correction	Potential interferences
6	Li	**I.S.**	-(0.0813)(^{7}C)	
7	Li	^{6}Li, Sc		
9	Be	^{6}Li, Sc		
10	B	^{6}Li, Sc		
11	B	^{6}Li, Sc		
27	Al	^{6}Li, Sc		
45	Sc	**I.S.**		CO_2H^+
47	Ti	^{6}Li, Sc		
49	Ti	^{6}Li, Sc		
51	V	^{6}Li, Sc	-(3.127)(^{53}C)+(0.352)(^{52}C)	$^{35}ClO^+$, $^{34}SOH^+$
52†	Cr	Sc, Y, Rh		ArC^+, ArO^+, $^{35}ClHO^+$
53‡	Cr	Sc, Y, Rh		$^{37}ClHO^+$
54†	Fe	Sc, Y, Rh	-(0.0284)(^{52}C)	
55	Mn	Sc, Y, Rh		$ArNH^+$
56	Fe	Sc, Y, Rh		
57‡	Fe	Sc, Y, Rh		
58†	Ni	Sc, Y, Rh		
59	Co	Sc, Y, Rh		
60	Ni	Sc, Y, Rh		
62	Ni	Sc, Y, Rh		TiO
63†	Cu	Sc, Y, Rh		$^{31}PO_2^+$, $^{40}ArNa^+$, TiO
65‡	Cu	Sc, Y, Rh		TiO
66	Zn	Sc, Y, Rh		TiO
68	Zn	Sc, Y, Rh		
75	As	Y, Rh	-(3.132)(^{77}C)+(2.736)(^{83}C)	$^{40}Ar^{35}Cl^+$
76	$^{40}Ar^{36}Ar^+$	Y, Rh		$^{40}Ar^{37}Cl^+$
77	Se	Y, Rh		
78†	Se	Y, Rh	-(0.1869)(^{76}C)‡	$^{40}Ar^{38}Ar^+$
82‡	Se	Y, Rh		$^{81}BrH^+$
83	Kr	Y, Rh		
88	Sr	Y, Rh		
89	Y	**I.S.**		
90	Zr	Y, Rh		
95	Mo	Y, Rh		$^{79}BrO^+$
98	Mo	Y, Rh	-(0.146)(^{99}C)	$^{79}BrHO^+$
99	Ru	Y, Rh		
103	Rh	**I.S.**		
105	Pd	Rh		
106	Pd, Cd	Rh		ZrO,
107	Ag	Rh		ZrO
108	MoO	Rh		ZrO, MoO
109	Ag	Rh		ZrO, MoO
111	Cd	Rh		ZrO, MoO
112	Cd	Rh	-(0.040)(^{118}C)	ZrO, MoO
114	Cd	Rh	-(0.0269)(^{118}C)	MoO
118	Sn	Rh		
119	Sn	Rh		
120	Sn	Rh	-(0.0127)(^{125}C)	
121	Sb	Rh	-(0.124)(^{125}C)	$^{40}Ar^{81}Br^+$
123	Sb	Rh		
125	Te	Rh		
137	Ba	Rh		
138	Ba	Rh	-(8.91E-04)(^{139}C)-(2.82E-04)(^{140}C)	
139	La	Rh		
140	Ce	Rh		
185	Re	**I.S.**		
203	Tl	Re		
205	Tl	Re		
206	Pb	Re		
207	Pb	Re		
208	Pb	Re	+(1.0)(^{206}C)+(1.0)($^{2.07}$C)	

Notes:
- † Recommended for the PE instrument.
- ‡ Recommended for the HP instrument.

Table 6. Quality Control Items, Frequency, and Corrective Action.

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tuning	After warm-up. Every 12 hours.	Manufacturer specifications	Check operating parameters, clean cones, replace malfunctioning components if necessary. Reevaluate the tuning.
ICV	After initial calibration.	90-110%	Verify that method parameters are valid, check calibration tables, replace calibration standards if necessary, and recalibrate the instrument.
ICB	Following ICV.	<RL for water samples	Prepare fresh calibration blank and/or increase the rinse time between analyses; reanalyze ICB; if within limits, continue the run; if still outside limits, determine the source of the problem, make the necessary corrections, and start from the beginning with a new calibration.
BS	After initial calibration.	70-130%	Verify that method parameters are valid, check calibration tables, replace calibration standards if necessary, prepare a fresh calibration blank, and recalibrate the instrument.
CCV	Before and after each batch. Every 10 sample. After re-calibration.	90-110%	Recalibrate the instrument. Follow method specific requirements (6020 or 200.8) as to what data prior to the CCV can be used.
CCB	Following CCV.	<RL for water samples	Prepare fresh calibration blank; reanalyze CCB; if within limits, continue the run; if still outside limits, eliminate the source of the contamination, clean the sample introduction system if necessary, and recalibrate the instrument. Reanalyze all samples from the last good CCB.
LCS	Every batch of 20 samples.	85-115%	Re-digest the entire sample batch and reanalyze.
LRB	Every batch of 20 samples.	<RL for water samples	Re-digest the entire sample batch and reanalyze.
Dp	Every 10 samples.	0-20%	If all other QC acceptable continue the run; sample result should be flagged; otherwise recalibrate instrument and reanalyze samples.
MS	Every 10 samples, prior to digestion.	70-130% with 200.8 75-125% with 6020	For 200.8 flag data if all other QC met; otherwise recalibrate instrument and reanalyze affected samples. For 6020 dilute original sample, re-spike this dilution, and reanalyze until within limits.
MSD†	Every 10 samples, prior to digestion.	0-20%	Same as for duplicate.
Dil‡	Every batch of 20 samples.	0-10%	If concentration analyzed >100 x MDL, flag data for possible matrix interference.
ICS-A‡	Every 12 hours.	<RL for water samples	Reevaluate the equations used for corrections, make the necessary adjustments, and recalibrate the instrument.
ICS-AB‡	Every 12 hours.	70-135%	Reevaluate the equations used for corrections, make the necessary adjustments, and recalibrate the instrument.
IS	With every analysis.	60-125% with 200.8 30-120% samples with 6020 80-120% for CCB with 6020	For samples, dilute 4+1 and reanalyze until in control. For CCV/CCB's recalibrate the instrument and reanalyze the affected samples.

NOTE:
- RL = Reporting Limit.
- Dil = Dilution Test.
- † MSD optional instead of duplicate sample.
- ‡ When Method 6020 referenced in the analytical report.

Appendix 1.

ICP/MS STANDARDS PREP LOG

Date	Standards	**IS id**	**ExpIRATION Date**	Analyst

Source: Inorganic Venture (IV-7 + IV-19)

Lot #: _____

Exp. Date:_____

ICP/MS INITIAL CALIBRATION VERIFICATION (ICV) PREP LOG

Date	ICV ID	IS id	**EXPIRATION DATE**	Analyst

Source ID **Lot#** **Expiration Date**

S = Spex _____ _____ _____

H = High Purity _____

E = ERA _____

APPENDIX 2. METALS DIGESTION

DATE _____

ANALYST _____

PREP BATCH MTD_____

2. SAMPLE#	BTL ID	SAMPLE AMOUNT GRAMS (g)	FINAL VOLUME (ml)	REMARKS	% TOTAL SOLIDS	DILUTION FACTOR
LCS -	-- --	50	**50**		——	1
LRB-	-- --	50	**50**		——	1

NOTES: *1) Spike values (unless otherwise stated):*
LCS = 0.05 ppm = 50 mls / 0.25 mls of 10 ppm WS1

2.1. Samples: Water = 0.05 ppm = 50 mls / 0.25 mls of 10 ppm WS1

Soil = 0.10 ppm = 50 mls / 0.50 mls of 10 ppm WS1
WS 1 - Lot # Y-MEB194014 + Z-CICP18146

2) Spile values for minerals (Ca-Mg-K-Na)
LCS = 1.0 ppm = 50 mls / 0.50 mls HP Stock Solution
Samples (Water or Soil) = 2.0 ppm = 50 mls / 1.0 mls HP Stock Solution
High Purity Stock Solution - Lot # 620719

3) HNO_3 Lot # 068109